

ADDENDUM

DRAFT FIELD OPERATIONS PLAN

Technical Assistance
for Galena Subsite Mine
Waste Characterization
Cherokee County Site
223-7L37/DEN67900.PM

S00022766
SUPERFUND RECORDS
2507

May 1988

INTRODUCTION

This addendum to the May 1988 Field Operations Plan (FOP) is for an additional field characterization and sampling episode. This sampling episode will be performed by the Galena PRP group in participation with the pilot testing program to be conducted in May 1989 at the Galena Subsite. CH2M Hill will provide project oversight for the sampling, and testing episodes, and will accept splits of PRP samples for submittal to the Region VII Laboratory.

This investigation will provide technical data to support design development of a portion of a possible remedy for the Groundwater and Surface Water Operable Unit. The method of remediation proposed for study in the PRP's testing program would include disposal of coarser-sized, low geochemically active mine waste rock mixed with chat containing lower zinc concentrations below the water table in the subsite's existing open mine voids. The more geochemically active, finer sized waste rock and contaminated chat would be deposited above the water table, in the open mine voids, to aid in backfilling some of the previously

mined areas. These materials would then be recontoured and covered with clean soils and chat to minimize infiltration and surface water capture.

This Addendum presents the sampling objectives, and sampling procedures including tentative locations. The Quality Assurance Project Plan (QAPP) for the Cherokee Site has also been revised for the this investigation.

All field oversight activities will be subject to a site specific Health and Safety Plan to provide for the safe execution of field activities.

Site access approvals will be obtained by either the PRP's or EPA prior to entering privately owned areas of the site.

SAMPLING OBJECTIVES

The objectives of this field sampling event are to characterize mine waste rock, chat, and groundwater and to select representative samples for use in the PRP's pilot testing program. Chemical and physical data obtained in this phase will allow development of the most appropriate combination of design and operating parameters for the geochemical batch and flow-through tests.

SAMPLING PROCEDURES, AND LOCATIONS

Due to the large areal extent of deposition and heterogeneous characteristics of the mine wastes (both rock and chat), it is very difficult to design an economical, statistically valid, bulk

mine waste sampling program. To compensate for this difficulty a parametric test method (varying design and operating parameters with waste rock type, and chat zinc grade) for the pilot test work will be developed.

Mine Waste Rock

Therefore, sample collection will be designed and reviewed, in the field, in order to collect representative silicious and calcareous waste rock samples based primarily on visual identification and mineralogy. A portable X-ray fluorescence (XRF) spectrometer will be used with reference standards of galena (PbS) and sphalerite (ZnS) for semiquantitative field measurements of chat materials.

Waste rock samples from the piles selected and sampled in the field will be transported in trucks from the sampling location to a central stockpile area at or near which the pilot testing will actually occur. Very large waste rock, say greater than 8" to 10" in diameter will be hand sorted and excluded since they begin to tax the size of the test apparatus and would have very little chemical impacts to the test program due to their low relative mineralized surface area. Separate stockpiles for siliceous and calcareous waste rock will be made and maintained.

Waste rock from each of the two stockpiles will be physically screened at a nominal plus 2 inches, and two stockpiles (+ 2 inch, and - 2 inch) developed for each rock type. The plus 2 inch material will be used in the subsequent pilot leach testing, but samples of both sized fractions will be taken for chemical analyses. A backhoe will be used to trench the graded stockpiles, forming a sampling sub-stockpile comprising of approximately one quarter of the total volume of the original plus and minus 2 inch stockpiles. These sub-stockpiles will be

similarly quartered and requartered until samples of approximately 200 pounds of each rock type and size are remaining.

The approximately 200 pound bulk samples will be stored in 5 gallon buckets with lids for transportation to the selected testing laboratories under chain of custody procedures.

At the primary laboratory, the samples will be sieved into approximately 5 to 6 grain-size samples each, both to produce data on grain-size distributions and to allow subsampling for chemical and mineralogical analyses. Each of the subsamples will be crushed and pulverized to minus 200 mesh, then replicate will be split for submittal for check analyses. Approximately 20 percent of the replicates will actually be submitted to the Region VII laboratory for lead, zinc, cadmium, and sulfur analyses. Additional samples may be selected by the PRP field team manager in consultation with the EPA oversight personnel for XRD analysis of mineralogy.

Chat

Chat selected on the basis of zinc concentration as determined in the field by use of the portable XRF unit and stockpiled at the central location. This material will be quartered and sampled similarly to the mine waste procedures. Approximately 200 pounds of chat will be transported to the laboratory for chemical analysis. The chat will be crushed and pulverized to minus 200 mesh, and replicate samples split for submittal to the Region VII Laboratory for lead, zinc, cadmium, and sulfur analyses.

Groundwater

To characterize the groundwater to be used in the pilot testing program, initial field observation and samples of groundwater from several mine void locations will be collected. Samples will be collected using a stainless steel or teflon bailer. Samples will initially be stored in properly labeled, inflatable cubitainers. As soon as possible the samples will be transferred to plastic bottles, filtered, and preserved as appropriate, and stored in insulated coolers under chain of custody procedures. A replicate bottle will be provided for submittal to Region VII Laboratory for RAS metals analyses. It is anticipated that about 20 percent of the replicate samples will be submitted for check analyses.

Mark
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see this

DRAFT
QUALITY ASSURANCE PROJECT PLAN
(QAPP)

CHEROKEE COUNTY SITE
Revision 2
GALENA SUBSITE MINE WASTE
CHARACTERIZATION PROGRAM

223-7L37/DEN67900

May 1989

This document has been prepared for the U.S. Environmental Protection Agency under Contract No. 68-01-7251. The material contained herein is not to be disclosed to, discussed with, or made available to any person or persons, for any reason without the prior express approval of a responsible official of the U.S. Environmental Protection Agency.

DE/CC11/044.1

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DRAFT
Quality Assurance Project Plan
For
Cherokee County Site
REVISION 2

Prepared for U.S. Environmental Protection Agency

Prepared by
CH2M HILL
EPA WA NO. 223-7L37
CH2M HILL PROJECT NO. DEN67900

MAY 1989

Section 1
INTRODUCTION

Environmental Protection Agency (EPA) policy requires all Remedial Investigation/Feasibility Study (RI/FS) activities to be under the control of a centrally managed quality assurance (QA) program. This requirement applies to all environmental monitoring and measurement efforts mandated or supported by EPA.

Parties generating data have the responsibility to implement procedures that assure that the precision, accuracy, completeness, comparability, and representativeness of their data are known and documented. EPA requires that a Quality Assurance Project Plan (QAPP) be developed to document the QA aspects to be used in performing the RI/FS. The QAPP presents, in specific terms, the policies, organization, objectives, functional activities, and specific QA and quality control (QC) activities to be used during implementation of the RI/FS activities.

This QAPP was prepared using EPA's Guidance for Preparation of Combined Work/Quality Assurance Project Plans for Environmental Monitoring (EPA, May 1984) and CH2M HILL's REM IV Zone Management Plan (CH2M HILL, May 1986). Other documents pertinent to QA/QC activities at the site include the following documents:

Work Plan, Remedial Investigation, and Feasibility Study, Cherokee County, Galena Subsite, June 10, 1985.

Final Quality Assurance Project Plan (QAPP), Cherokee County, Galena Subsite, July 24, 1985.

Draft Laboratory Analytical Protocol for Groundwater, Surface Water, Soil, Sediment, and Fish Samples, Cherokee County, Galena Subsite July 26, 1985.

Laboratory Analytical Protocol for Air Quality, Cherokee County, Galena Subsite, August 7, 1985.

Final Draft, Phase I Remedial Investigation Report, Cherokee County, Galena Subsite, April 23, 1986.

Work Plan and FOP, Site-Wide Surface Hydrology Program, Low Flow Conditions, September 12, 1986.

Final Work Plan, Site-Wide Water Supply Inventory, Cherokee County Site, March 6, 1987.

Final Work Plan, Surface and Subsurface Hydrology Investigations, Spring 1987, Cherokee County Site and Galena Subsite, March 9, 1987.

Work Plan, Supplementary Remedial Investigations for the Galena Subsite Mine Wastes Characterization and Geophysics, May 1987.

Technical Memorandum, Water Quality of Groundwater in the Galena Area, June 1987.

Technical Memorandum, Water Quality of Surface Water in the Cherokee County Site, July 1987.

Technical Memorandum, Sitewide Water Supply Inventory, November 1987.

Final Draft, Alternate Water Supply Operable Unit Feasibility Study, Galena Subsite, November 1987.

Final Draft, Groundwater and Surface Water Operable Unit Feasibility Study, Galena Subsite, February 1988.

Technical Memorandum, Ground Penetrating Radar and Spontaneous Potential Surveys in the Galena Area, February 1988.

Activity-specific operations are discussed in these documents. Reference will be made to these documents throughout this QAPP, and appropriate documents such as the SOP will be included as appendices to this QAPP.

This QAPP is intended to be a functional document for all onsite and offsite personnel involved with activities related to field operations at the Cherokee County site. Field activities and their procedures are identified in detail in an effort to help onsite personnel properly follow Region VII and CH2M HILL protocol.

This QAPP/SOP is intended to be a document that can be revised as the project evolves. Additional sections will be added to the SOP as the project expands or as new methodologies are required. Also, sections of the QAPP can be revised as needed. The format of this QAPP/SOP and its loose-leaf binding will facilitate periodic updates. Each page is identified with a page-specific number (section, page, revision) so individual pages or entire sections can be replaced or revised. Each copy of the QAPP/SOP will be serially numbered and a distribution list of all copies will be maintained by the SM.

QAPP, Section 1
Revision No. 1
Date: 5/31/88
Page: 3 of 3

All proposed revisions to the Final QAPP/SOP will be submitted first to the SM and RPM for review and comment. The proposed change(s) will then be approved by the SM, RPM, and Regional VII QA Officer. The SM will prepare all revisions so they can be inserted into the QAPP/SOP by exchanging new pages for existing pages, and then send the revised pages to all the individuals who have been assigned a copy of the Project QAPP.

Section 2 BACKGROUND INFORMATION

This section presents a brief summary of existing data related to the site. The site's physical setting, history, and related contamination events are discussed. For more detailed information concerning the site, refer to the documents listed in Section 1.

PHYSICAL SETTING

The Cherokee County site is a triangular-shaped area in the southeast corner of Kansas and includes the communities of Treece, Baxter Springs, and Galena (see Figure 2-1). Portions of Cherokee County, Kansas; Ottawa County, Oklahoma; and Jasper County, Missouri comprise what is known as the Tri-State Mining District. Earlier in this century, the District had one of the richest lead-zinc ore deposits in the world. The entire Tri-State mining area encompasses approximately 500 square miles. Because of the large size of the Cherokee County Site (about 100 square miles), it has been divided into six subsites, based on the presence of physical surface effects of abandoned lead and zinc mining (see Figure 2-1). Collectively, these subsites have a total area of approximately 25 square miles.

The EPA Region VII directed initial RI activities to focus on the Galena subsite, an approximately 9-square-mile area in the east-central portion of the Cherokee County Site (Figure 2-1). The QAPP for the Galena Subsite RI was prepared in July 1985 (EPA, 1985c). The current QAPP is concerned with the Sitewide Surface and Subsurface Hydrology Investigations, the Sitewide Water Supply Inventory/Sampling program, and the field investigations that may precede the OUFS's at Galena for the groundwater and surface water systems. This QAPP will also be used for additional remedial investigations during 1987 and 1988 and will be revised as necessary for these future efforts. This revision includes the 1988 Spring-Summer mine waste sampling and survey program in support of development of the proposed remedy for the Galena Subsite Groundwater and Surface Water Operable Unit (EPA 1988a).

SITE HISTORY

Underground mining operations in the Cherokee County Site area ranged from depths of approximately 50 feet to almost 400 feet. The area is honeycombed with mines. Water was pumped continually from these mines during the active mining

years. When mining activity and pumping from the mines ceased, the mines filled quickly with water through natural groundwater recharge and direct inflow of surface runoff into mine

Section 3 PROGRAM OBJECTIVES

The principal concern at the Cherokee County site is acid mine drainage (AMD) into the area's surface streams and shallow groundwater. This AMD contains relatively high concentrations of heavy metals such as zinc, lead, and cadmium and because of its low pH, is corrosive. The source of AMD is the sulfide minerals that occur naturally in the rock strata throughout the Tri-State Mining District, and these same minerals that exist as residual ores left in the mine workings and in mine wastes at the surface (such as chat piles and tailings ponds). The sulfide minerals are the source; the groundwater and surface water systems are the main transport mechanisms.

The program objectives (EPA 1988b) are basically to:

1. Determine representative "high" and "low" grade mine waste (chat/waste rock) characteristics through field X-ray Fluorescence (XRF) measurements at the Galena Subsite.
2. Collect samples that represent the high and low grades found on the Galena Subsite for the purpose of metallurgical evaluation designed for the selection of mine waste treatment equipment and operating parameters.
3. Conduct a survey of the mine waste types and pile dimensions to refine previous volume estimates of the surface mine wastes (waste rock and chat) at the Galena Subsite.

The remedial actions should fall into two major categories; first--those that will remove, contain, or isolate the source material, and second--those that would reduce the generation and transport of AMD (i.e., reduce the volume, mobility, or toxicity of the contaminants).

The overall project approach will address three environmental media: the sulfide minerals that are the source of the problem, and the surface water and groundwater systems that act as the transport mechanisms that carry the toxic metals to humans, animals, and plants. The sulfide minerals exist as residual ores in the mine wastes left at the site, and as natural minerals in the ground. In some subsites, air may also be an important transport mechanism and will be included in the RI/FS program. The contaminants of interest at the site are inorganic, and mostly the so-called heavy metals. Organic contaminants are not an issue at this site.

Section 4
QUALITY ASSURANCE OBJECTIVES

The overall purpose of establishing quality assurance objectives for measurement data is to ensure that data of known and acceptable quality are provided for the intended data use. The data generated by this project are to be of such quality that:

- o They can be used for determination of design and operating parameters required for development of a mine waste milling/flotation treatment unit for the Galena Subsite surface mine wastes.

The specific objectives of the quality assurance program are to:

- o Provide an estimate of analytical precision and accuracy of laboratory test results
- o Provide verification of the occurrence of contaminants
- o Provide data that estimate the high and low grade of metals (primarily lead and zinc) in the mine waste

To meet the overall and specific quality assurance objectives, the following QA/QC parameters will be addressed for all data measurements:

- o Completeness
- o Comparability
- o Representativeness
- o Accuracy
- o Precision

COMPLETENESS

Completeness is a measure of the amount of valid data that was obtained from a measurement system to the amount that

Table 7-1
RESPONSIBILITY MATRIX

<u>CH2M HILL Project</u> <u>Team Member and Position</u>	<u>Responsibilities</u>
Neil Geitner Site Manager	<ul style="list-style-type: none"> - Overall project management <ul style="list-style-type: none"> - Coordination with EPA RPM, EPA QA office, the RM, and ZPMO - Monthly status reports - Document development - QA/AC - Corrective actions - Financial and schedule control
Mike Thompson Regional Manager	<ul style="list-style-type: none"> - Coordination with EPA RPM, EPA QA office, and ZPMO - Expertise in Superfund sites - Review documents for QC
Bill Bluck Review Team Leader	<ul style="list-style-type: none"> - Coordination with RM and XPMO - Senior Technical Review - Expertise in mining sites - Technical planning and QC
Greg Peterson Quality Assurance Manager	<ul style="list-style-type: none"> - Review QA procedures and actions
Jerry May <u>Mine Waste Characterization</u> <u>Task Leader</u>	<ul style="list-style-type: none"> - <u>Conformance to Work Plan/QAPP</u> - <u>Field quality control</u> - <u>Field crew and equipment scheduling</u> - <u>Sample shipping</u> - <u>Maintain Field Logs</u>

DOCUMENT CONTROL

Document control requirements have been established in order to ensure:

- o Documents are distributed and released in accordance with EPA policy
- o Documents are kept secure and under custody where necessary. Controls will be maintained to prevent unauthorized reproduction and/or alteration
- o Document holders are known
- o Document holders receive report revisions and updates when appropriate (for example, the SOP will require periodic revisions)

Document control requirements have been divided into two categories: tracking and distribution.

TRACKING

Document tracking will conform to the requirements of QAMS-005/80 (EPA, 1980). Although QAMS-005/80 designates tracking procedures for QAPP's only, the intent of these procedures will be followed for all project deliverables.

Project files will be maintained in accordance with Document Control Procedures, Denver Remedial Planning Center.

Each project report, deliverable, or document kept in the project files is assigned a specific document identification number. The document number is constructed by using a combination of the work assignment number, office location, project log number, and a consecutive series number. For example:

Document No. is 102.7L37.O.D.B7-2.005m

where: 102.7L37.0 = EPA Work Assignment Number

D = Denver office

B7-2 = Project Document Log Number

005 = Serial number from 001--999 to identify the first work plan from the second and third, etc.

m = Serial code from a--z to identify each copy of the same report

A distribution list is maintained for each deliverable submitted to EPA, and that list includes the document number(s) sent to each person.

APPENDIX A. STANDARD OPERATING PROCEDURES

CONTENTS (Revision 1, 5/31/88)

	<u>Page</u>
0 Introduction	0-1
1 Equipment Calibration, Operation and Maintenance	1-1
1.1 Introduction	1-1
1.2 QA Program Policies as Applied to Equipment	1-1
1.3 Calibration and Field Check Schedule	1-1
1.4 pH Meter	1-3
1.5 Electrical (Specific) Conductivity Meter	1-5
1.6 Kemmerer or Van Dorn Specific-Depth Samplers	1-7
1.7 Dissolved Oxygen Meters	1-8
1.8 Flow Meters	1-9
1.9 Temperature Measurement	1-11
2 Establishing Stream Transects and Selecting Sampling Verticals	2-1
3 Calculation of Cross-Sectional Area	3-1
4 Open-Channel Flow Measurements	4-1
4.1 Wading in the Stream Channel Flow Measurement	4-1
4.2 Bridge Deck or Boat Flow Measurement	4-4
5 Temperature and Conductivity Profiles and Dissolved Oxygen	5-1
6 Composite Stream Sampling	6-1
6.1 Wading in Stream Water Quality Sample	6-1
6.2 Bridge or Boat Water Quality Sample	6-3
6.3 Composite Water Quality Sample	6-4
7 Filtration of Dissolved Metals Samples	7-1
8 Decontamination Procedures	8-1
8.1 Personnel	8-1
8.2 Sample Bottles and Sampling Equipment	8-1
8.3 Field Vehicles	8-2
8.4 Disposal of Materials Generated Onsite	8-2
9 Sample Numbering System	9-1
10 Sample Quantities, Bottles, and Preservatives	10-1

CONTENTS (continued)

	<u>Page</u>
11 Packing and Shipping Procedures	11-1
12 Instructions for Filling Out Documentation	12-1
12.1 Requesting Analytical Services	12-1
12.2 General Documentation Procedures	12-4
12.3 Sample Identification Matrix	12-6
12.4 Completing Contract Laboratory Program Forms	12-8
12.5 EPA Region VII Sample Labels	12-25
12.6 EPA Region VII Field Sheet	12-25
12.7 Receipt for Samples Form	12-29
12.8 Field Log Book	12-30
12.9 Corrections to Documentation	12-31
12.10 Laboratory Custody	12-31
13 Groundwater Monitoring	13-1
13.1 Water Level Measurements	13-1
13.2 Water Sampling	13-1
14 Radon Sampling-Water	14-1
15 <u>X-Ray Fluorescence Spectrometer</u>	<u>15-1</u>

Section 15
X-RAY FLUORESCENCE SPECTROMETER

FIELD ANALYTICAL TECHNIQUES--X-RAY
FLUORESCENCE SPECTROMETER

The chemical characterization of Quality Assurance/Quality Control (QA/QC) mine waste material in the field is proposed to be performed by the field portable x-ray fluorescence (XRF) spectrometer ATX-100 instrument manufactured by Aurora Tech, Inc, 331 Rio Grande Street, Salt Lake City, Utah. The instrument uses low level self-contained and shielded radioactive sources that produce spectrum of peaks which position (energy level) is specific to an individual element and peak height which is indicative of the concentration of that element within the area exposed to the source. Two sources are proposed to be used, cadmium-109 (15 millicuries) and americium-241 (19 millicuries) implaced by the manufacturer. The cadmium source will allow semiquantitative determination of the copper, zinc, arsenic, and lead concentrations. Additional elements that will be monitored include chromium, manganese, iron, cobalt, nickel, selenium, and molybdenum. The americium source will be used for the semiquantitative determination of cadmium and barium concentration.

Additional elements that will be monitored with the americium source include silver, tin, and antimony.

The detection limit for the instrument is a function of source strength, geometry/particle size, counting time, and the element concentration. Since the source strength and instrument geometry are constants, the detection limit is dependent on geometry/particle size, counting time, and concentration. It has been demonstrated that 80 mesh particle size dominantly composed of a siliceous or caleareous skeletal matrix will give analytical results within 20 percent. The larger the particle size, the larger the error. On a large mass the

larger the particles, the larger the error--a rock made up of fine-grained minerals, however, will essentially have the same precision and accuracy as a finely ground sample.

The counting time also affects the detection limit. In general, the longer the counting time, the lower the detection limit, and certainly the higher the precision and accuracy. The instrument has controllable time units of 10, 30, 100, 300, and manual control seconds. The 100 second counting time will likely be the standard for this test. This time should be low enough to x-ray pyrite as well as limestone and not exceed a total count level that will affect accuracy and precision. This rate may change for either or both sources depending on the actual sample matrix encountered in the field.

Experience with similar instruments with poorer resolution indicate that the semiquantitative detection limit can be expected to be below 10 parts per million on the five elements of primary concern. One to two parts per million is achievable but not with high precision. A suite of archived analyzed sample splits from earlier sampling of the tailings and soils will be used for calibration. They will be used to calculate a precision, accuracy, and detection limit. The samples will be scanned and the measurements recorded each day before the instrument is used.

The primary operator will receive one day's training on the proper use of the instrument particularly for health and safety purposes. The manufacturer's statement on radiation safety is also attached. The primary operator has over 5 years experience using similar instrument in field applications analogous to this application. The operator will have the dates and time used logged in the record book specifically kept for this purpose.

RADIATION SAFETY

Aurora Tech is licensed by the Utah Bureau of Radiation Control as an agreement state under the authority of the Nuclear Regulatory Commission.

The ATX-100 is manufactured under License UBRC-1800030 and is sold under the General License UBRC-1800031. This general license grants Aurora Tech the right to manufacture and sell the ATX-100 to unlicensed purchasers.

The ATX-100 user has available three sources for use in the sensor head, although only two will be installed in any one sensor head. The specifications of these sources are as follows:

	<u>Fe-55</u>	<u>Cd-109</u>	<u>Am-241</u>	<u>Units</u>
Initial Source Strength	100	15	19	mCi
Primary X-ray Energy	5.9	22.2	60	keV
Other Radiation	None	88 keV	Alpha	
		Gamma Ray		
Half-Life	2.6	1.2	458	Years
Service Life	3-10	2-5	800+	Years

The radioactive sources employed in the device are sealed by the supplier and are leak tested before and after their insertion into the sensor head. It is extremely unlikely that Aurora Tech would ever sell an instrument with a leaking source of radioactivity. The purchaser has the subsequent responsibility for periodic leak tests. Any possible contamination would be identified by these tests.

According to measurements made by the Utah Bureau of Radiation Control under the authority of the Nuclear Regulatory Commission, the dose rate with the shutter closed at 5 cm. (about 2 inches) is about 4.1 mrem/hr.. and at 30 cm. (about 1 foot) it is 1.15 mrem/hr. A radiation worker is allowed 5,000 mrem/yr. total body dose and about 30,000 mrem/yr to the skin (each and every year). Therefore, a person would have to remain 2 inches from the sensor head for $(30,000 \text{ mrem} / 4.1 \text{ mrem/hr.} =) 7,317$ hours or 305 days of 24 hours each or $(5,000 \text{ mrem} / .015 \text{ mrem/hr.} = 33,333 \text{ hours})$ about 1,389 days of 24 hours each to accumulate his allowable annual dose. Obviously, it is doubtful that anyone would remain 2 inches away from the sensor head for 305 24-hour days and since there are not 1,389 days in the year, total body dose rate will not be exceeded.

Purposeful and biological significant exposure by a person by the radiological sources in the sensor head is possible, but this event seems quite unlikely since it would take a considerable time to accomplish. Because of the severe collimation of the direct radiation beam emerging from the instrument head, a total body exposure is impossible at a short distance. Also, it is not possible for a person to stand at more than an arm's length from the sensor head and still keep the shielding pulled back against the spring. This arm's length position would result in less than a total body exposure with the sensor head close to the body. The resulting maximum exposure rates might be about 6,000 mrem/hr. In order to accumulate 30 rem, the exposure time would have to be 5 hours. If the location of the exposure was not constant, the total dose received by any one volume of tissue would be greatly reduced. Therefore, a biological significant and purposeful, let alone inadvertent, exposure seems unlikely.

Exposure hazard from the ATX-100 is extremely small. This is because of the instrument design, quality control measures in manufacture, and the safety procedures inherent in ruggedness testing and the proper operation by suitable trained purchasers. Film safety badges will be used by the sample team for additional protection as described in the Health and Safety Plan.

Appendix B
ANALYTICAL METHODS AND DETECTION LIMITS

Revision 1
Cherokee County Site
223-7L37/DEN67900

CONTENTS

	<u>Page</u>
Laboratory Analytical Protocol	B-1
General Description	B-1
Services Requested	B-1
Sample Collection, Preservation, and Holding Times	B-1
Analytical Methods	B-1
Sample Concentration	B-5
Sample Shipments	B-5
Laboratory Holding Times and Reporting Times	B-5
Reporting Format	B-5
Report Transmittal	B-7
Laboratory QC	B-7
Archive Requirements	B-9
<u>Metallurgical Test Work</u>	<u>B-10</u>

TABLES

1	Sample-Services Summary Sheet Cherokee County Site, Galena Subsite	B-2
2	Sample Size Preservation and Holding Time	B-3
3	Analytical Methods Summary Sheet Cherokee County Site, Galena Subsite	B-4
4	Analytical Methods and Detection Limits Cherokee County Site, Galena Subsite	B-6
5	Laboratory QA/QC Requirements for Water Chemistry Parameters Cherokee County Site and Galena Subsite	B-8

METHOD 1310

EXTRACTION PROCEDURE (EP) TOXICITY TEST METHOD AND STRUCTURAL INTEGRITY TEST

1.0 SCOPE AND APPLICATION

1.1 This method is employed to determine whether a waste exhibits the characteristic of Extraction Procedure Toxicity.

1.2 The procedure may also be used to simulate the leaching which a waste will undergo if disposed of in a sanitary landfill. Method 1310 is applicable to liquid, solid, and multiphase samples.

2.0 SUMMARY OF METHOD

2.1 If a representative sample of the waste contains $>0.5\%$ solids, the solid phase of the sample is ground to pass a 9.5 mm sieve and extracted with deionized water which is maintained at a pH of 5 ± 0.2 , with acetic acid. Wastes that contain $<0.5\%$ solids are not subjected to extraction but are directly analyzed. Monolithic wastes which can be formed into a cylinder 3.3 cm (dia) x 7.1 cm, or from which such a cylinder can be formed which is representative of the waste, may be evaluated using the Structural Integrity Procedure instead of being ground to pass a 9.5-mm sieve.

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Extractor: For purposes of this test, an acceptable extractor is one that will impart sufficient agitation to the mixture to (1) prevent stratification of the sample and extraction fluid and (2) ensure that all sample surfaces are continuously brought into contact with well-mixed extraction fluid. Examples of suitable extractors are shown in Figures 1-3 of this method and are available from: Associated Designs & Manufacturing Co., Alexandria, Virginia; Glas-Col Apparatus Co., Terre Haute, Indiana; Millipore, Bedford, Massachusetts; and Rexnord, Milwaukee, Wisconsin.

4.2 pH meter or pH controller: Accurate to 0.05 pH units with temperature compensation.

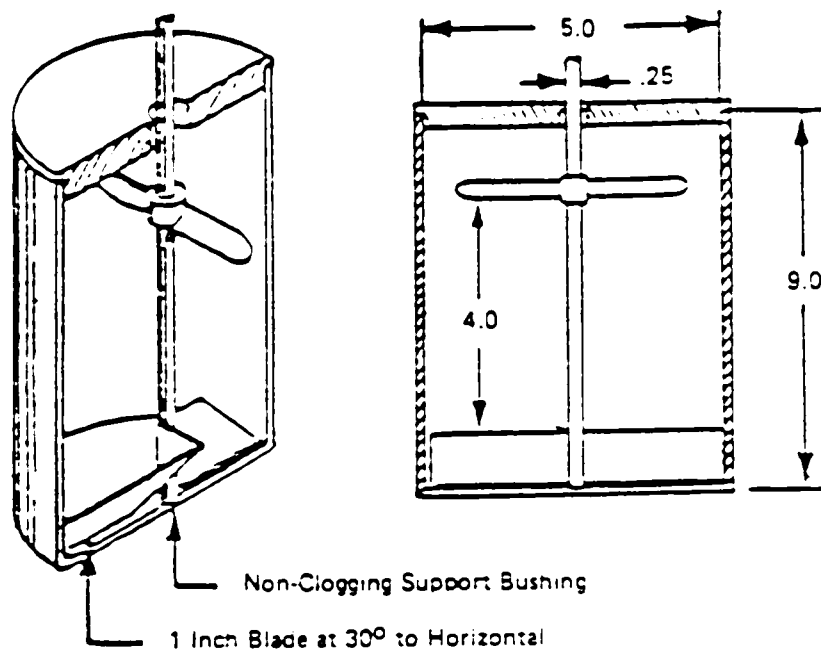


Figure 1. Extractor.

2 - Liter Plastic or Glass Bottles

1/15 - Horsepower Electric Motor

29 RPM

Screws for Holding Bottles

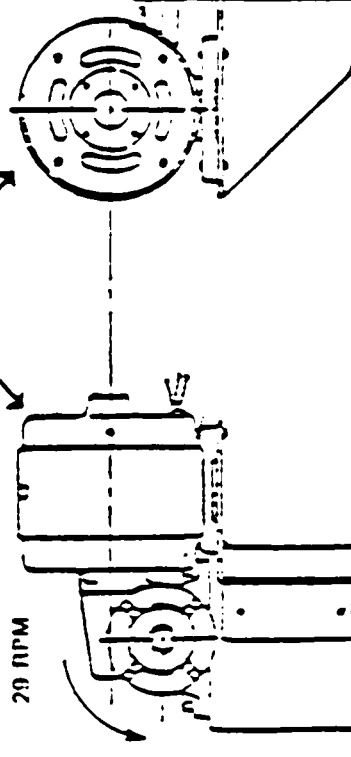


Figure 2. Rotary Extractor.

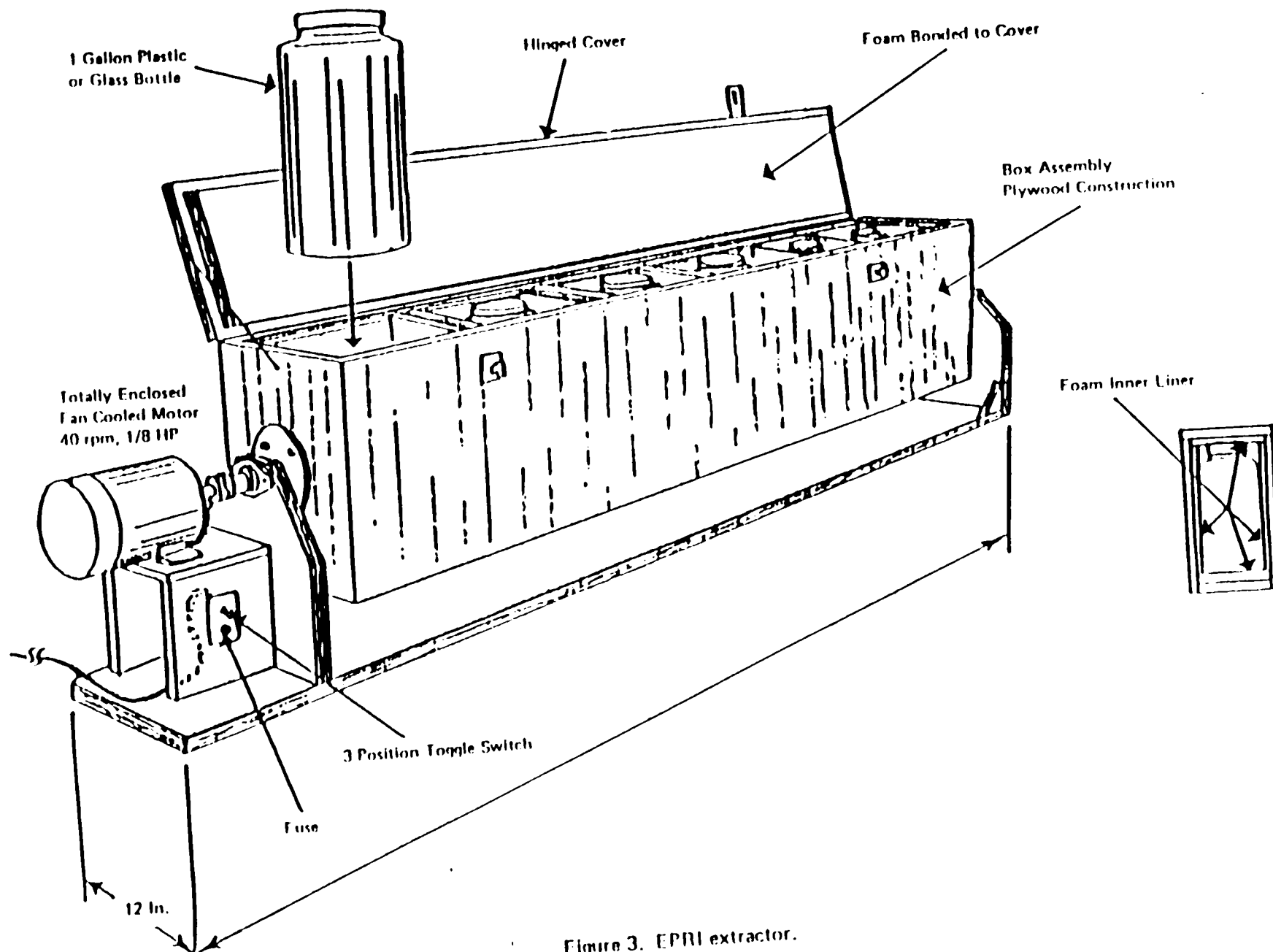


Figure 3. EPRI extractor.

1310-4

Revision 0
Date September 1966

4.3 Filter holder: Capable of supporting a 0.45-um filter membrane and of withstanding the pressure needed to accomplish separation. Suitable filter holders range from simple vacuum units to relatively complex systems that can exert up to 5.3 kg/cm³ (75 psi) of pressure. The type of filter holder used depends upon the properties of the mixture to be filtered. Filter holders known to EPA and deemed suitable for use are listed in Table 1.

4.4 Filter membrane: Filter membrane suitable for conducting the required filtration shall be fabricated from a material that (1) is not physically changed by the waste material to be filtered and (2) does not absorb or leach the chemical species for which a waste's EP extract will be analyzed. Table 2 lists filter media known to the agency to be suitable for solid waste testing.

4.4.1 In cases of doubt about physical effects on the filter, contact the filter manufacturer to determine if the membrane or the prefilter is adversely affected by the particular waste. If no information is available, submerge the filter in the waste's liquid phase. A filter that undergoes visible physical change after 48 hr (i.e., curls, dissolves, shrinks, or swells) is unsuitable for use.

TABLE 1. EPA-APPROVED FILTER HOLDERS

Manufacturer	Size	Model No.	Comments
<u>Vacuum Filters</u>			
Nalgene	500 mL	44-0045	Disposable plastic unit, including prefilter, filter pads, and reservoir; can be used when solution is to be analyzed for inorganic constituents.
Nuclepore	47 mm	410400	
Millipore	47 mm	XX10 047 00	
<u>Pressure Filters</u>			
Nuclepore	142 mm	425900	
Micro Filtration Systems	142 mm	302300	
Millipore	142 mm	YT30 142 HW	

TABLE 2. EPA-APPROVED FILTRATION MEDIA

Supplier	Filter to be used for aqueous systems	Filter to be used for organic systems
<u>Coarse prefilter</u>		
Gelman	61631, 61635	61631, 61635
Nuclepore	210907, 211707	210907, 211707
Millipore	AP25 035 00, AP25 127 50	AP25 035 00, AP25 127 50
<u>Medium prefilters</u>		
Nuclepore	210905, 211705	210905, 211705
Millipore	AP20 035 00, AP20 124 50	AP20 035 00, AP20 124 50
<u>Fine prefilters</u>		
Gelman	64798, 64803	64798, 64803
Nuclepore	210903, 211703	210903, 211703
Millipore	AP15 035 00, AP15 124 50	AP15 035 00, AP15 124 50
<u>Fine filters (0.45 um)</u>		
Gelman	60173, 60177	60540 or 66149, 60544 or 66151
Pall	NX04750, NX14225	
Nuclepore	142218	142218 ^a
Millipore	HAWP 047 00, HAWP 142 50	FHUP 047 00, FHLP 142 50
Selas	83485-02, 83486-02	83485-02, 83486-02

^aSusceptible to decomposition by certain polar organic solvents.

4.4.2 To test for absorbtion or leaching by the filter:

4.4.2.1 Prepare a standard solution of the chemical species of interest.

4.4.2.2 Analyze the standard for its concentration of the chemical species.

4.4.2.3 Filter the standard and reanalyze. If the concentration of the filtrate differs from that of the original standard, then the filter membrane leaches or absorbs one or more of the chemical species and is not usable in this test method.

4.5 Structural integrity tester: A device meeting the specifications shown in Figure 4 and having a 3.18-cm (1.25-in.)-diameter hammer weighing 0.33 kg (0.73 lb) with a free fall of 15.24 cm (6 in.) shall be used. This device is available from Associated Design and Manufacturing Company, Alexandria, VA 22314, as Part No. 125, or it may be fabricated to meet these specifications.

5.0 REAGENTS

5.1 Acetic acid (0.5 N): This can be made by diluting concentrated glacial acetic acid (17.5 N) by adding 57 mL glacial acetic acid to 1,000 mL of water and diluting to 2 liters. The glacial acetic acid should be of high purity and monitored for impurities.

5.2 Analytical standards should be prepared according to the applicable analytical methods.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

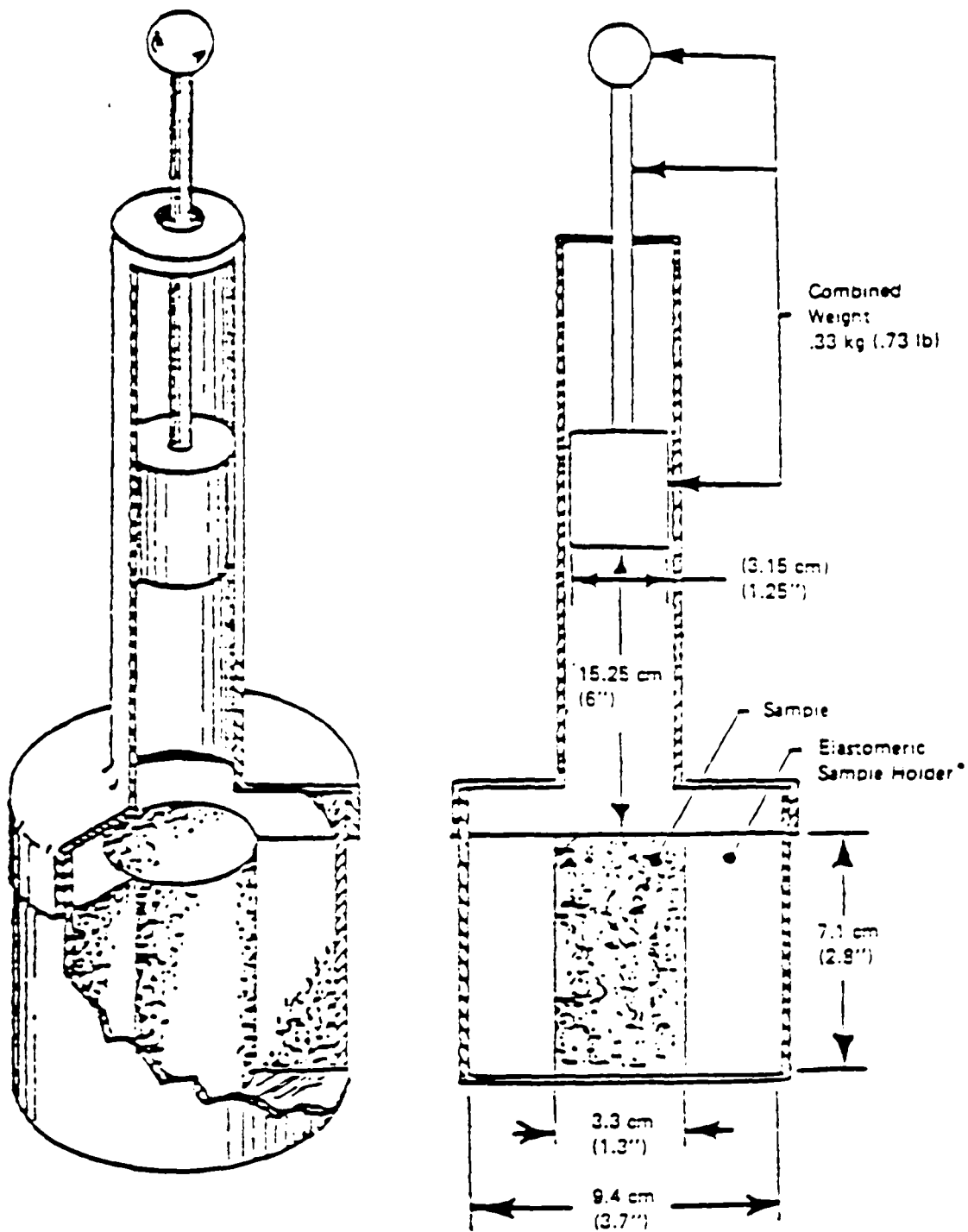
6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 Preservatives must not be added to samples.

6.3 Samples can be refrigerated if it is determined that refrigeration will not affect the integrity of the sample.

7.0 PROCEDURE

7.1 If the waste does not contain any free liquid, go to Step 7.9. If the sample is liquid or multiphase, continue as follows. Weigh filter membrane and prefilter to ± 0.01 g. Handle membrane and prefilters with blunt curved-tip forceps or vacuum tweezers, or by applying suction with a pipet.



* Elastomeric sample holder fabricated of material firm enough to support the sample.

Figure 4. Compaction tester.

7.2 Assemble filter holder, membranes, and prefilters following the manufacturer's instructions. Place the 0.45-um membrane on the support screen and add prefilters in ascending order of pore size. Do not prewet filter membrane.

7.3 Weigh out a representative subsample of the waste (100-g minimum).

7.4 Allow slurries to stand, to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.5 Wet the filter with a small portion of the liquid phase from the waste or from the extraction mixture. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10-15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10-psi increments to 75 psi. Halt filtration when liquid flow stops. This liquid will constitute part or all of the extract (refer to Step 7.16). The liquid should be refrigerated until time of analysis.

NOTE: Oil samples or samples containing oil are treated in exactly the same way as any other sample. The liquid portion of the sample is filtered and treated as part of the EP extract. If the liquid portion of the sample will not pass through the filter (usually the case with heavy oils or greases), it should be carried through the EP extraction as a solid.

7.6 Remove the solid phase and filter media and, while not allowing them to dry, weigh to ± 0.01 g. The wet weight of the residue is determined by calculating the weight difference between the weight of the filters (Step 7.1) and the weight of the solid phase and the filter media.

7.7 The waste will be handled differently from this point on, depending on whether it contains more or less than 0.5% solids. If the sample appears to have $< 0.5\%$ solids, determine the percent solids exactly (see Note below) by the following procedure:

7.7.1 Dry the filter and residue at 80°C until two successive weighings yield the same value.

7.7.2 Calculate the percent solids, using the following equation:

$$\frac{\text{weight of filtered solid and filters} - \text{tared weight of filters}}{\text{initial weight of waste material}} \times 100 = \% \text{ solids}$$

NOTE: This procedure is used only to determine whether the solid must be extracted or whether it can be discarded unextracted. It is not used in calculating the amount of water or acid to use in the extraction step. Do not extract solid material that has been dried at 80°C . A new sample will have to be used for extraction if a percent solids determination is performed.

7.8 If the solid constitutes $<0.5\%$ of the waste, discard the solid and proceed immediately to Step 7.17, treating the liquid phase as the extract.

7.9 The solid material obtained from Step 7.5 and all materials that do not contain free liquids should be evaluated for particle size. If the solid material has a surface area per g of material $>3.1 \text{ cm}^2$ or passes through a 9.5-mm (0.375-in.) standard sieve, the operator should proceed to Step 7.11. If the surface area is smaller or the particle size larger than specified above, the solid material is prepared for extraction by crushing, cutting, or grinding the material so that it passes through a 9.5-mm (0.375-in.) sieve or, if the material is in a single piece, by subjecting the material to the "Structural Integrity Procedure" described in Step 7.10.

7.10 Structural Integrity Procedure (SIP):

7.10.1 Cut a 3.3-cm-diameter by 7.1-cm-long cylinder from the waste material. If the waste has been treated using a fixation process, the waste may be cast in the form of a cylinder and allowed to cure for 30 days prior to testing.

7.10.2 Place waste into sample holder and assemble the tester. Raise the hammer to its maximum height and drop. Repeat 14 additional times.

7.10.3 Remove solid material from tester and scrape off any particles adhering to sample holder. Weigh the waste to the nearest 0.01 g and transfer it to the extractor.

7.11 If the sample contains $>0.5\%$ solids, use the wet weight of the solid phase (obtained in Section 7.5) to calculate the amount of liquid and acid to employ for extraction by using the following equation:

$$W = W_f - W_t$$

where :

W = wet weight in g of solid to be charged to extractor;

W_f = wet weight in g of filtered solids and filter media; and

W_t = weight in g of tared filters.

If the waste does not contain any free liquids, 100 g of the material will be subjected to the extraction procedure.

7.12 Place the appropriate amount of material (refer to Step 7.11) into the extractor and add 16 times its weight of Type II water.

7.13 After the solid material and Type II water are placed in the extractor, the operator should begin agitation and measure the pH of the solution in the extractor. If the pH is >5.0 , the pH of the solution should be decreased to 5.0 ± 0.2 by adding 0.5 N acetic acid. If the pH is ≤ 5.0 , no

acetic acid should be added. The pH of the solution should be monitored, as described below, during the course of the extraction, and, if the pH rises above 5.2, 0.5 N acetic acid should be added to bring the pH down to 5.0 ± 0.2 . However, in no event shall the aggregate amount of acid added to the solution exceed 4 mL of acid per g of solid. The mixture should be agitated for 24 hr and maintained at 20-40°C (68-104°F) during this time. It is recommended that the operator monitor and adjust the pH during the course of the extraction with a device such as the Type 45-A pH Controller, manufactured by Chemtrix, Inc., Hillsboro, Oregon 97123, or its equivalent, in conjunction with a metering pump and reservoir of 0.5 N acetic acid. If such a system is not available, the following manual procedure shall be employed.

7.13.1 A pH meter should be calibrated in accordance with the manufacturer's specifications.

7.13.2 The pH of the solution should be checked, and, if necessary, 0.5 N acetic acid should be manually added to the extractor until the pH reaches 5.0 ± 0.2 . The pH of the solution should be adjusted at 15-, 30-, and 60-min intervals, moving to the next longer interval if the pH does not have to be adjusted >0.5 pH units.

7.13.3 The adjustment procedure should be continued for at least 6 hr.

7.13.4 If, at the end of the 24-hr extraction period, the pH of the solution is not below 5.2 and the maximum amount of acid (4 mL per g of solids) has not been added, the pH should be adjusted to 5.0 ± 0.2 and the extraction continued for an additional 4 hr, during which the pH should be adjusted at 1-hr intervals.

7.14 At the end of the extraction period, Type II water should be added to the extractor in an amount determined by the following equation:

$$V = (20)(W) - 16(W) - A$$

where:

V = mL Type II water to be added;

W = weight in g of solid charged to extractor; and

A = mL of 0.5 N acetic acid added during extraction.

7.15 The material in the extractor should be separated into its component liquid and solid phases in the following manner:

7.15.1 Allow slurries to stand to permit the solid phase to settle (wastes that are slow to settle may be centrifuged prior to filtration) and set up the filter apparatus (refer to Steps 4.3 and 4.4).

7.15.2 Wet the filter with a small portion of the liquid phase from the waste or from the extraction mixture. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10-15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10-psi increments to 75 psi. Halt filtration when liquid flow stops.

7.16 The liquids resulting from Steps 7.5 and 7.15 should be combined. This combined liquid (or waste itself, if it has <0.5% solids, as noted in Step 7.8) is the extract and should be analyzed for the presence of any of the contaminants specified in 40 CFR Part 261.24 using the analytical procedures as designated in Step 7.17.

7.17 The extract is then prepared and analyzed using the appropriate analytical methods described in Chapters 3 and 4 of this manual.

NOTE: If the EP extract includes two phases, concentration of contaminants is determined by using a simple weighted average. For example: An EP extract contains 50 mL of oil and 1,000 mL of an aqueous phase. Contaminant concentrations are determined for each phase. The final contamination concentration is taken to be:

$$\frac{(50)(\text{contaminant conc. in oil}) + (1,000)(\text{contaminant conc. of aqueous phase})}{1050}$$

7.18 The extract concentrations are compared with the maximum contamination limits listed in 40 CFR Part 261.24. If the extract concentrations are greater than or equal to the respective values, the waste then is considered to exhibit the characteristic of Extraction Procedure Toxicity.

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 All quality control measures described in Chapter 1 and in the referenced analytical methods should be followed.

9.0 METHOD PERFORMANCE

9.1 The data tabulated below were obtained from records of state and contractor laboratories and are intended to show the precision of the entire method (1310 plus analysis method).

TABLE 3. PRECISIONS OF EXTRACTION-ANALYSIS
PROCEDURES FOR SEVERAL ELEMENTS

Element	Sample Matrix	Analysis Method	Laboratory Replicates
Arsenic	1. Auto fluff	7060	1.8, 1.5 ug/L
	2. Barrel sludge	7060	0.9, 2.6 ug/L
	3. Lumber treatment company sediment	7060	28, 42 mg/L
Barium	1. Lead smelting emission control dust	6010	0.12, 0.12 mg/L
	2. Auto fluff	7081	791, 780 ug/L
	3. Barrel sludge	7081	422, 380 ug/L
Cadmium	1. Lead smelting emission control dust	3010/7130	120, 120 mg/L
	2. Wastewater treatment sludge from electroplating	3010/7130	360, 290 mg/L
	3. Auto fluff	7131	470, 610 ug/L
	4. Barrel sludge	7131	1100, 890 ug/L
	5. Oil refinery tertiary pond sludge	7131	3.2, 1.9 ug/L
Chromium	1. Wastewater treatment sludge from electroplating	3010/7190	1.1, 1.2 mg/L
	2. Paint primer	7191	61, 43 ug/L
	3. Paint primer filter	7191	--
	4. Lumber treatment company sediment	7191	0.81, 0.89 mg/L
	5. Oil refinery tertiary pond sludge	7191	--
Mercury	1. Barrel sludge	7470	0.15, 0.09 ug/L
	2. Wastewater treatment sludge from electroplating	7470	1.4, 0.4 ug/L
	3. Lead smelting emission control dust	7470	0.4, 0.4 ug/L
Lead	1. Lead smelting emission control dust	3010/7420	940, 920 mg/L
	2. Auto fluff	7421	1540, 1490 ug/L
	3. Incinerator ash	7421	1000, 974 ug/L
	4. Barrel sludge	7421	2550, 2800 ug/L
	5. Oil refinery tertiary pond sludge	7421	31, 29 ug/L

(Continued)

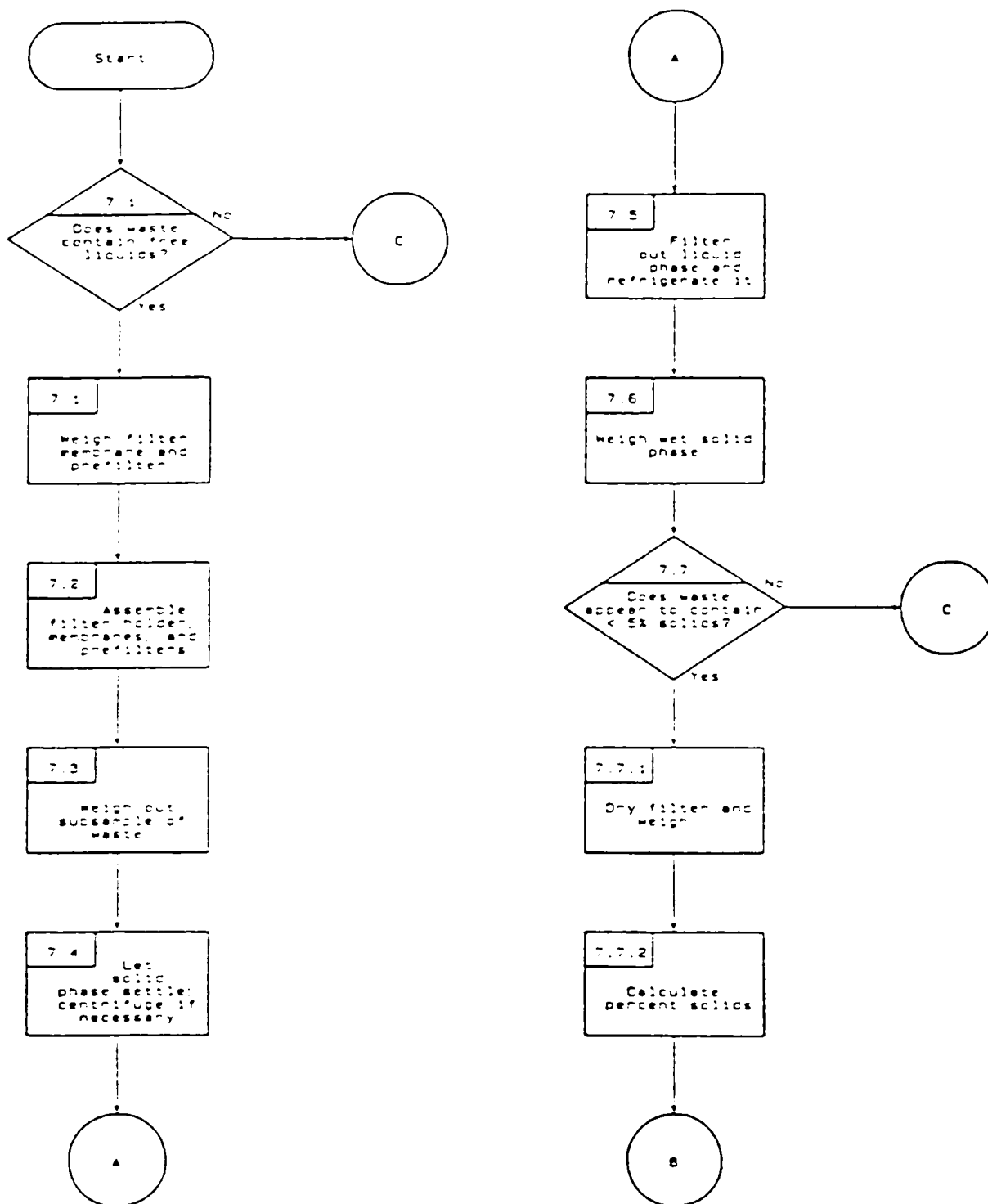
TABLE 3 (Continued)

Element	Sample Matrix	Analysis Method	Laboratory Replicates
Nickel	1. Sludge	7521	2260, 1720 ug/L
	2. Wastewater treatment sludge from electroplating	3010/7520	130, 140 mg/L
Chromium(VI)	1. Wastewater treatment sludge from electroplating	7196	18, 19 ug/L

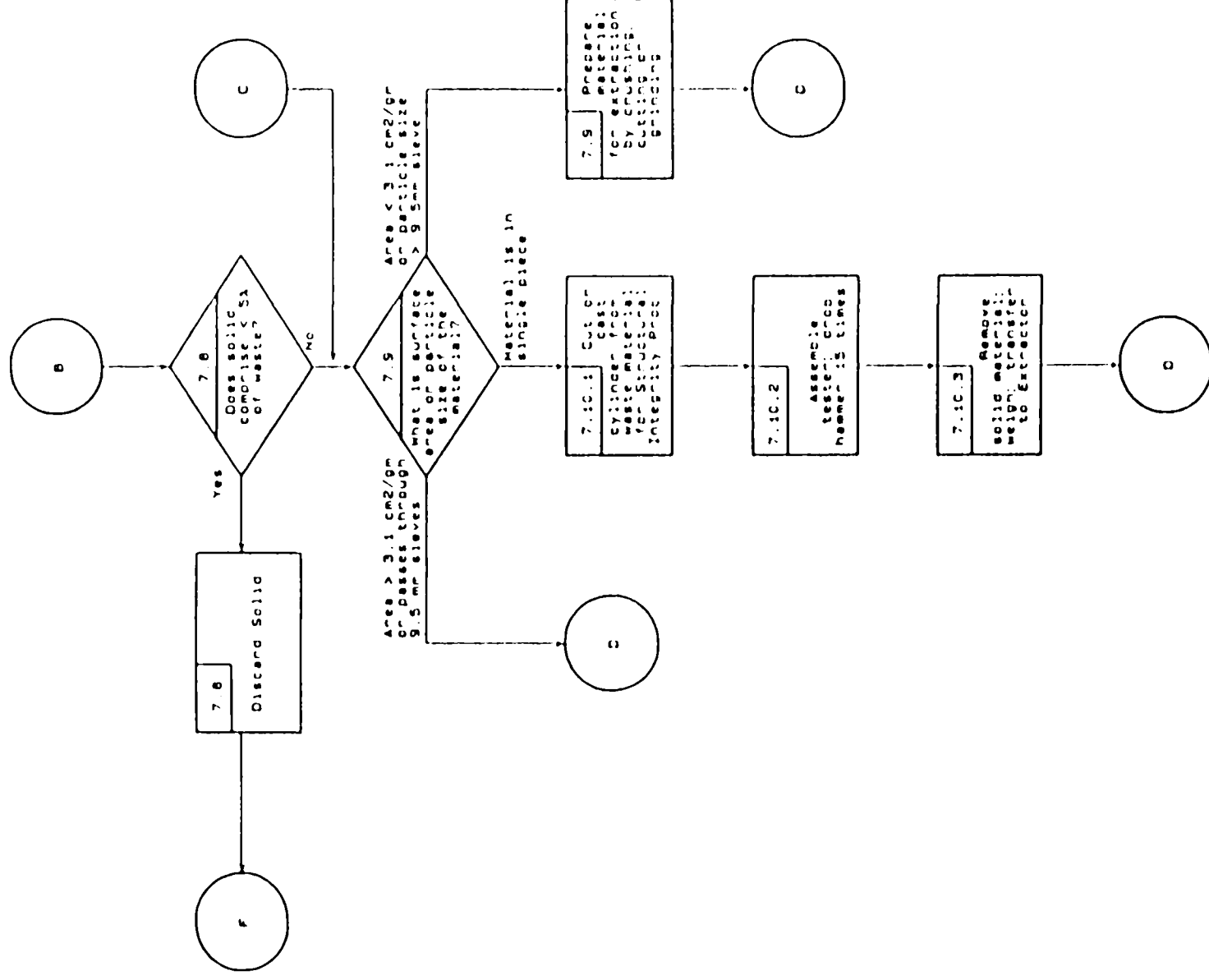
10.0 REFERENCES

1. Gaskill, A., Compilation and Evaluation of RCRA Method Performance Data, Work Assignment No. 2, EPA Contract No. 68-01-7075, September 1986.

Method 1310
EXTRACTION PROCEDURE (EP) TOXICITY TEST METHOD
AND STRUCTURAL INTEGRITY TEST



METHOD 1310
EXTRACTION PROCEDURE (EPI) TOXICITY TEST METHOD
AND STRUCTURAL INTEGRITY TEST
(Continued)



METHOD 1310
EXTRACTION PROCEDURE (EP) TOXICITY TEST METHOD
AND STRUCTURAL INTEGRITY TEST
(Continued)

